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STUDY OF BRONZE POROUS ALLOY Cu-Sn WORKED OUT BY METALLUGY OF THE POWDERS

F. Keraghel[a,b]* , k. Loucif[a], M. P. Delplancke[b] *

[a]*LMNM, DOMP, Fac des Sces de l'Ingénieur, Univ. F.A. 19000 Sétif, Algérie*

[b]***L.M.M. Fac. des Scies Appliquées, Université Libre de Bruxelles, Belgique***

Abstract

Porous bronzes take popularity in various fields of technology.
Their development is based on the metallurgy of powders. The samples, in the present study, are worked out by pressure sintering pressure. We used various techniques of characterization: density, hardness, optical and electronic microscopy and diffraction of x-rays. We showed that in the temperature and pressure range or field swept the density believes linearly with these two parameters. The secondary phase was identified. By microscopy, we proved that the structure is not homogeneous.

Keywords : Bronze, sintering, porosity, mapping, antifriction ;

1. INTRODUCTION

Being an advanced material either in mechanics or in electronics, the making of porous bronze is a much studied subject. Elements of additions (tin, lead, iron, aluminum) were added in order to improve the mechanical resistance and the behavior to wear [1-8]. Porous bronzes are frequently worked out by forming followed by a sintering in liquid phase [2].

The purpose of this work is to work out porous bronzes by pressure sintering. It aims then at determining the physical and microstructural characteristics of samples and to seek consequently the effect of development parameters (temperature, pressure) on these characteristics.

* Corresponding author. Tel.: 00-213-36-92-51-34; fax: 00-213-36-92-51-34.
E-mail address: f_keraghel@yahoo.fr

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2. EXPERIMENTAL METHODS

2.1. Materials

The tin and copper powders are of great purity (99,98%) and a granulometry lower than 40 μm. The composition of our alloy is fixed at Cu-8%Sn. This choice is taken after a first study related to the compositions Cu-2%Sn, Cu-5%Sn and Cu-10%Sn where we notice that the addition of tin improves the mechanical properties, in particular hardness, but this last light marks a low for a content of 10%Sn [9]. The proportioning of the powders was carried out by means of a balance of precision to digital posting "Sartorius" of an error of measurement 0,1mg. The mixing of the powders has been carried out in a revolving earthenware jar at a speed of 100trs/min during 6 hours. Alumina balls were added in proportion 1:1 with the powder in order to ensure a homogeneous powder (ball milling).

2.2. Experimental techniques for the characterization

Characteristics of the samples concerning the measurement of the density and hardness. For the microstructural analysis, we used optical and electronic microscopy and the X-rays diffraction. Density is determined by the method of Arthur [9].

The Rockwell ball steel hardness of diameter 1.5mm was carried out on a durometer of GALILEO type, they were carried out with an initial load P_0 = 15N followed by a P_1 load= 150N during 10 second.

The observation techniques used are optical microscopy and electronic scan microscopy. The preparation of the porous samples requires a detailed attention. The samples are initially ground with an abrasive paper 800 in order to remove the oxide coating formed on the surface, and then successively last with a paper 1200, 2400, and 4000. The completion is made with a claw with diameter of 3 microns then a fraction of 1 micron.

The samples are then chemically attacked by a solution made up of 5 G of iron chloride ($FeCl_2$), 2cc of hydrochloric acid (HCI) and 95cc of ethanol alcohol. The duration of the attack is about a few seconds, and then the samples are cleaned and rinsed.

For the observation in electronic microscopy, we used an electronic scan microscope of the type Joel JSM - 6100 equipped with a detector of secondary electrons, a detector of retrodiffused electrons and a system of qualitative analysis of elements.

The analyses were carried out on a diffractometer SIEMENS D 5000 equipped with a copper tube (□=0,15406 nm) using DIFFRAC plus and TOPAS V2.0 softwares for the calculation of cell parameters.

3. EXPERIMENTAL RESULTS

3.1 *Study of the density*

Figure 1 gives the variation of the density according to the temperature under a pressure of 50MPa and for various duration periods. It is noticed that the density increases in a quasilinear way in the indicated field of temperature. Since the melting point of tin is about 320°C, it seems to us that the combined effect of the temperature and the pressure contributes to a sintering in liquid phase. Thus, as the temperature increases, tin wets well the grains of copper and playing the role of a binder, it facilitates the thickening of alloy. In addition, the increase in the temperature activates the couple of diffusion copper-tin.

The curves giving the variation of density with pressure for a sintering temperature of 250 ° C and different holding times are summarized in Figure 2. By analogy with those in Figure 1, they also show that in the area scanned, the density increases linearly with pressure. This increase can be explained by the rearrangement of the powder for low pressure values after settling. For high values of pressure, the contact surfaces expand, powder grains, being ductile, plastically deform to densify the compact and thus reduce the rate of porosity.

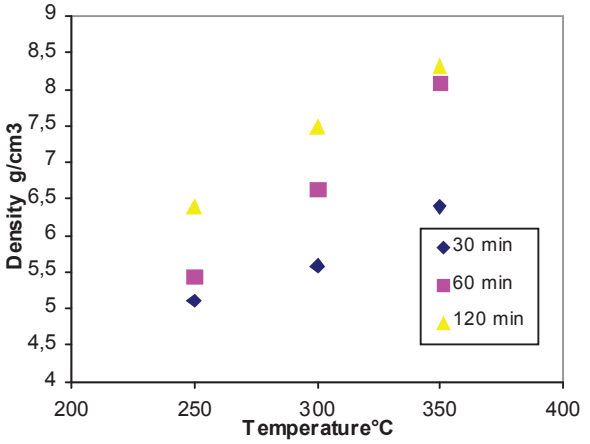


Fig. 1: Density variation according to sintering temperature under a pressure of 50 MPa and several durations of maintain

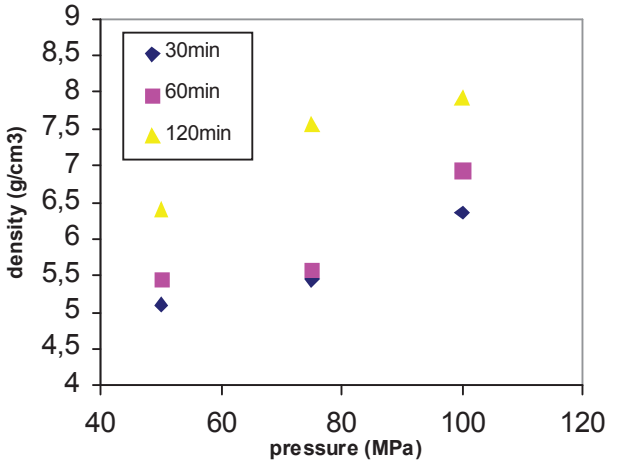


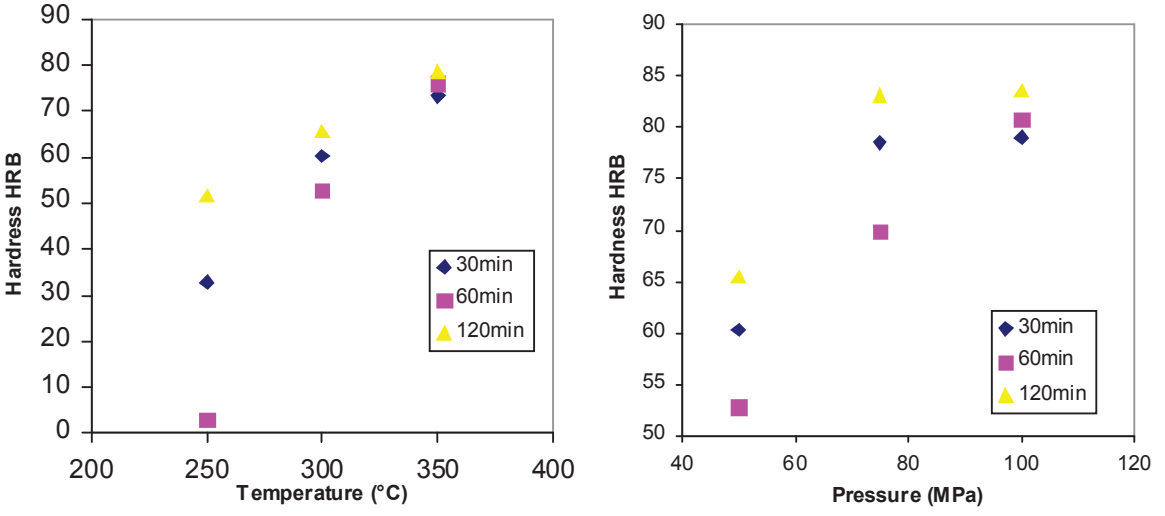
Fig. 2: Variation of the density according to the pressure for a temperature of sintering of 250°C and several durations of maintain.

3.2. *Study of hardness*

The variation of Rockwell ball hardness according to the temperature (with a pressure of P=50MPa) and of the pressure (at temperature of T=300°C) is presented by figure 3. Generally, the hardness of the samples increases when the temperature or/and the pressure increases. It is quite obvious that the increase in the temperature and the pressure grows the density of material consequently; this last proves a certain penetration resistance of the ball. However we cannot correlate this increase to a clear relation.

Micrograph (fig. 4) shows two quite distinct zones with a good contrast:

- a red zone, formed of dendrites , corresponding to the electrolytic copper particles,
- a very fine grayish zone corresponding to the tin particles obtained by atomization.



A : P= 50 MPa b : T= 300°C

Fig. 3: the hardness variation according to (a) the temperature and (b) pressure.

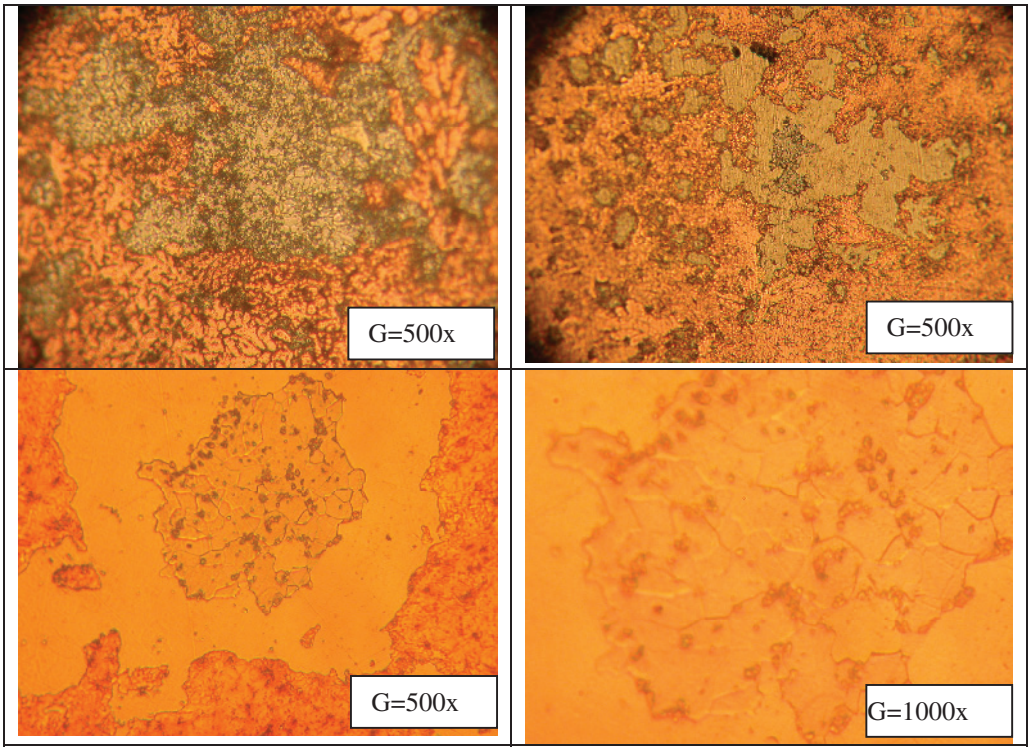


Fig. 4: Optical micrographs of the worked out sample with T=250°C, P=75MPa, t = 30 min

This micrography shows that the grains of the two powders remain in their starting states and no morphological transformation was noticed. It seems that the duration period is insufficient to guarantee a significant physicochemical connection between copper and tin. However, simultaneous action of temperature and the pressure led to a thickening of the mixture

Electronic micrography 5 confirms a heterogeneous structure similar to that given by optical micrographies made up of:

- zones rich in dendritic copper of structure when the temperature of sintering is low,
- zones rich in tin of form various depending on the temperature and the pressure of sintering,
- intermediate zones between those rich in copper and rich in tin and which correspond to the zones of the couple of diffusion coppers - tin,
- a porosity generalized especially with the areas low in tin where damping is weak.

The tests of cartography on MEB show very particular phases with composition proving the existence of composed except balance or metastable (fig. 6).

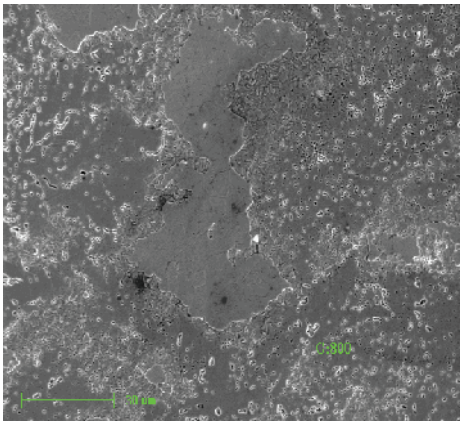
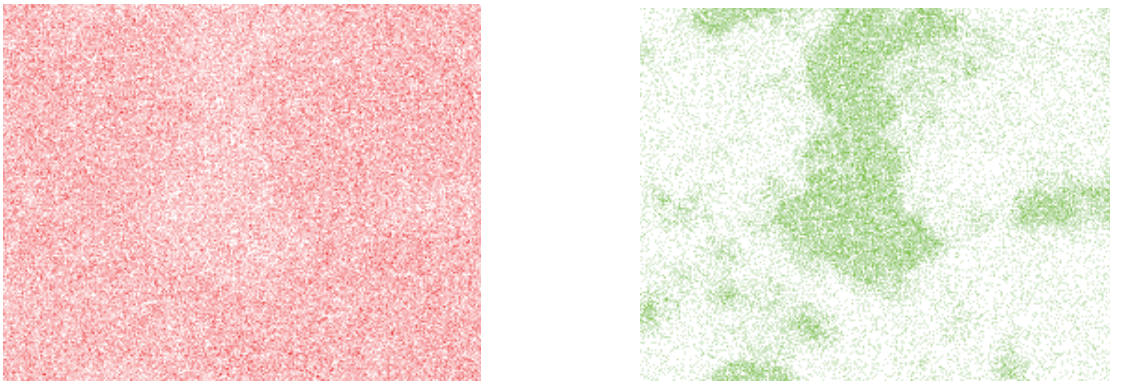


Fig. 5: Electronic micrography of alloy worked out under the conditions: T=350°C, P=50MPa, t=30min



a : Mapping of copper b : Mapping of tin

Fig. 6: Mapping Image of the area (Fig. 5) of alloy worked out from under the conditions:
T = 350 ° C, P = 50MPa, T = 30min

3.4. Study of the spectrum of the X-ray diffraction

We presented the spectra of X-ray diffraction according to the temperature of sintering in order to examine the effect of this parameter on the formation of the phase in question. Thus, we notice that at low temperature 250°C that no secondary phase takes form in a significant way. On the contrary, when the temperature increases the

intensities of the peaks corresponding to the secondary phases develop (fig. 7). By the analysis of X-ray diffraction, we manage to identify the following phases:
-the unalloyed copper, which did not react with the tin content of copper content significantly from that of tin,
-phase Cu_3Sn which is a stable phase and mainly formed for different parameters of development. It is orthorhombic structure resulting from the diffusion of tin in copper [2]. This phase confirmed by EDAX microanalysis of the phase gives the atomic ratio Cu / Sn = 74.31/24.23.
- the phases Cu_xSn_y type that can form under special conditions and are generally intermediate or metastable phases. Previous studies have shown that these types of phases are transformed into Cu3Sn after annealing [10].

We must point out that at the end of traces of copper oxides, CuO or Cu_2O type, were detected for long periods of sintering. These tracks, though still small, confirming the results of mapping by electron microscopy. However, peaks on the tin was not found.

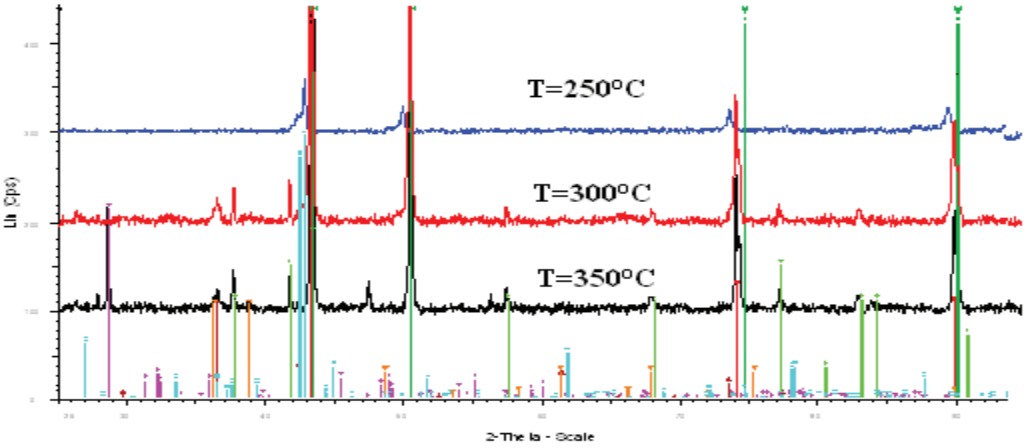


Fig. 7: Spectra of X-ray diffraction of samples prepared under a pressure of P = 50 MPa for t = 30 min and different temperatures.

4. CONCLUSION

- increase in the density function of processing parameters (temperature, pressure) and is almost linear in the fields scanned,

- hardness increases generally according to the parameters of development but we cannot find a relation clear taking into account the dispersions met especially in measurements of microhardness,

- determination of a secondary phase majority Cu_3Sn with the possibility of forming other secondary phases which are intermediate,
- the micrographic observation shows a heterogeneous structure of alloy with a generalized porosity

References

[1] A. P. Savitskii , N. N. Burtsev, and M. A. Emel'yanova, Dilatometric investigation of the processes of liquid phase sintring of materials of the Cu-Sn system, Poroshkovaya Metallurgiya, No. 9 (261)1984, pp.27-31
[2] N. N. Acharya, Effect of probe pressure during dilatometric studies on sintering of copper-tin bronzes, Journal of Materials Science Letters 18 (1999) pp681-683
[3] C. Alves, V. Hajek, C.A. dos Santos, Thermal behavior of supersolidus bronze powder compacts during heating by hollow cathode discharge, Materials Science and Engineering A348 (2003) pp84-89
[4] V. Kumar, Porous metal bearing – a critical review, Wear, 63 (1980) pp271-287

[5] D. Bouvard, G. Cizeron, métallurgie des poudres, Article de la Revue de Métallurgie C.I.T/Science et Génie des matériaux, (1994) 684p
[6] G. Parallis, G. Mader, Precis de Métallurgie, Edition dépôt égale, Paris (1990) 186 P.
[7] R. Cazaud, R. Leroux, Métallurgie mise en forme et traitements, Edition Dunod Paris (1951) 573p.
[8] S. Klpakgian, Manufacturing process for ingeneering materials, Edition Addison Wesley Massachusett (1984) 839p.
[9] F. Keraghel, Elaboration et caractérisation des bronzes frittés sous pression, mémoire de Magister, Institut de Mécanique, Université B. M. Annaba – Algérie (1999) 98p
[10] N.N. Acharya, P.G. Mukunda, Sintring in the copper-tin system : Identification of phases and reactions, The International Journal of Powder Metallurgy, V31, 1(1995) pp.63-72